88. Photolytic Reactions of (*o*-Allylbenzyl)dicarbonyl(η^5-cyclopentadienyl)iron

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Photolysis of (*o*-allylbenzyl)dicarbonyl(η^5-cyclopentadienyl)iron (**4**) at 20° in CH_2Cl_2 leads to carbon-monoxide loss followed by intramolecular complexation to [η^2-(*o*-allylbenzyl)]carbonyl(η^5-cyclopentadienyl)iron (**13**). At 50° in C_6D_6, a photochemical rearrangement proceeds forming carbonyl (η^5-cyclopentadienyl){η^3-[3-(2-methylphenyl)allyl]}iron (**17**). Depending on the temperature, photolysis of **4** leads to intermolecular reactions at the benzylic or allylic position of the π complexes **13** and **17**, respectively.

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1. Introduction. – Recently, we had shown that photolysis of benzyliron complex **1** leads to homolytic cleavage of the Fe—C bond and generates benzyl radicals (**2**). Reaction in the presence of electron-poor alkenes **3** yields C—C bond formation products [1] (*Scheme 1*).

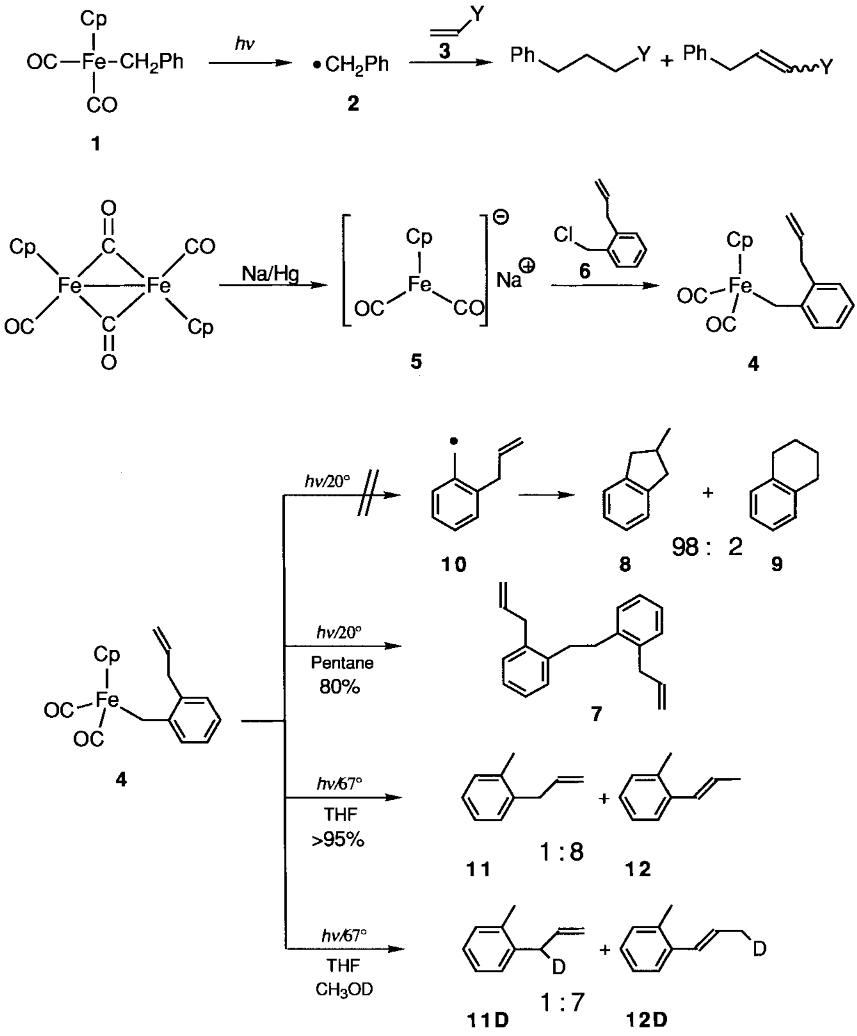
We have now tried to trap benzyl radicals intramolecularly by a suitably located C=C bond. Therefore, the allylbenzyl complex **4** was synthesized by treatment of the readily available iron salt **5** [2] with *o*-allylbenzyl chloride (**6**) [3]. Surprisingly, photolysis of complex **4** at 20° in pentane yielded exclusively bibenzyl derivative **7**. Cyclization products **8** and **9**, which are expected from intramolecular reaction of the *o*-allylbenzyl radical (**10**) [3], could not be detected. Photolysis of **4** at 67° in THF led almost quantitatively to reduction products **11** and **12** in a 1:8 ratio. In the presence of CH_3OD, D was incorporated in both allylic positions giving **11D** and **12D** in a 1:7 ratio.

These unexpected results led us to a more thorough investigation of the properties of the iron complex **4** under photolytic conditions.

2. Results and Discussion. – Irradiation of **4** at 20° in CH_2Cl_2 led within a few min quantitatively to the formation of π-coordinated species **13** (*Scheme 2*). The rearrangement was monitored by ^{1}H-NMR spectroscopy. The yield of isolated olefin complex **13** was 95%. A comparable intramolecular substitution reaction has already been reported by *Green* and *Smith* [4].

Compared to the complex **4**, the NMR data of π complex **13** show characteristic differences (*Table 1*). π Coordination of an alkene by a metal center usually leads to an upfield shift of the signals of the olefinic system, indicating increasing sp^3 character of the C-atoms of the C=C bond. It is also known that the signals of the H-atoms that are oriented towards the metal center are strongly influenced. Therefore, both the upfield shift of all olefinic and benzylic H-atoms of **13** and the decreased coupling constants of H_E—C(10) and H_Z—C(10) indicate π coordination of the C=C bond. The considerable

Scheme 1



Cp = Cyclopentadienyl

Scheme 2

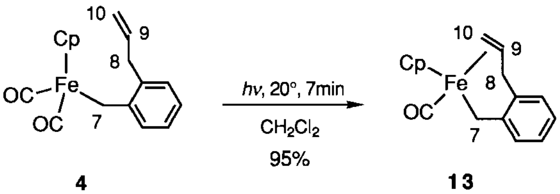


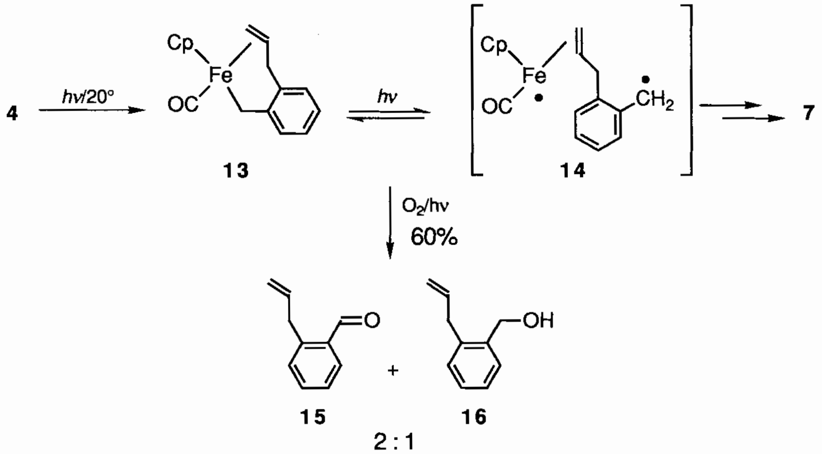
Table 1. *^{1}H- and ^{13}C-NMR Data for Iron Complexes* **4** *and* **13** (see *Scheme 2*)

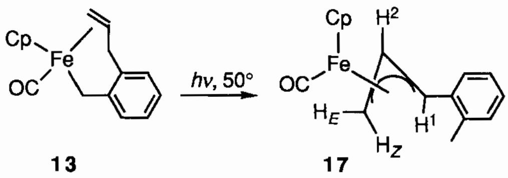
	^{1}H-NMR (C_6D_6)			^{13}C-NMR ($CDCl_3$)	
	4	**13**		**4**	**13**
H_a–C(7)	2.86	1.66	C(7)	0.4	1.0
H_b–C(7)		2.78			
H_a–C(8)	3.65	2.69	C(8)	37.4	34.0
H_b–C(8)		3.17			
H–C(9)	6.25	4.48	C(9)	138.0	37.9
H_E–C(10)	5.23 (J = 10)	3.00 (J = 8.5)	C(10)	115.5	5.5
H_Z–C(10)	5.26 (J = 17)	1.31 (J = 13)			

upfield shifts of the signals of C(9) and C(10) of *ca.* 100 ppm in the ^{13}C-NMR spectrum are even more indicative of π-complexation.

Further irradiation of π complex **13** at 20° might cleave the Fe–C σ bond, leading to a hypothetical intermediate which could have structure **14** of a cage biradical **14** (*Scheme 3*). Recombination yields bibenzyl derivative **7** after decomplexation. Intramolecular trapping of the benzylic radical part of **14** by the olefinic bond does not occur, because the C=C bond of **14** is still coordinated. However, the benzylic radical center of **14** could be trapped by O_2. Thus, photolysis of **4** at 20° in C_6D_6 in the presence of O_2 yielded aldehyde **15** and alcohol **16**.

Scheme 3





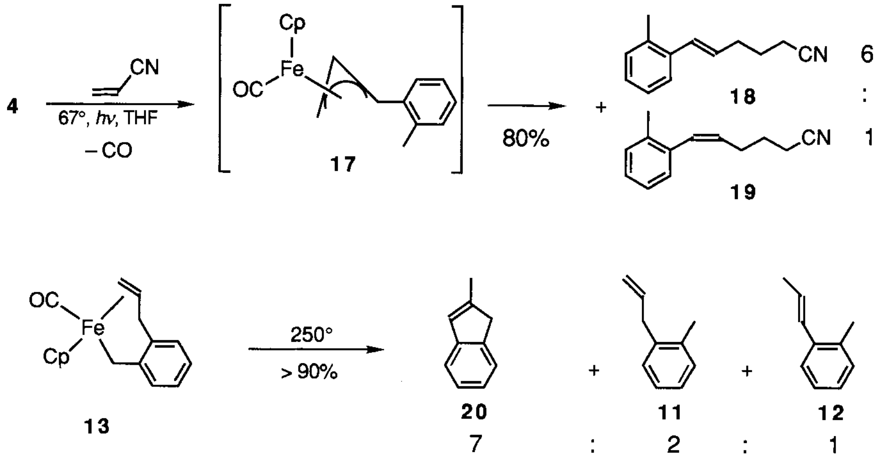
Irradiation of the π complex **13** at 50° in C_6D_6 caused within a few min a rearrangement to π-allyl complex **17**. The structure of **17** was determined by ^{1}H-NMR spectroscopy (*Table 2*). The upfield shifts of H_E, H_Z, and H^1 indicate an increasing sp^3 character of C(1) and C(3) of the coordinated allylic system. The value of the chemical shift of H^2 at around 5 ppm is typical of η^3-allyl complexes [1] [5]. The coupling constants (> 10 Hz) between H^1, H^2, and H_Z indicate a *trans*-configuration of π-allyl complex **17**. The *singlet* at 2.52 ppm is assigned to the aryl-substituted Me group of **17**.

Table 2. *^{1}H-NMR Data for η^3-Allyl Complex* **17** (C_6D_6; see *Scheme 3*)

H-Atom	δ [ppm]	*Multiplicity*	J [Hz]
H^1	2.19	*d*	10.7
H^2	5.15	*ddd*	11.2, 10.7, 6.8
H_E	2.85	*dd*	6.8, 1.5
H_Z	0.88	*dd*	11.5, 1.5
$ArCH_3$	2.52	*s*	
Cp	4.13	*s*	
Ar	6.9–7.5	*m*	

The formation of deuterated products **11D** and **12D** (*Scheme 1*) in THF/CH_3OD obviously occurs *via* reaction of CH_3OD with η^3-allyl complex **17** at both ends of the allylic system. Attack at the sterically less hindered unsubstituted allylic position yields the main product **12D**. To gain deeper insight into the reaction, we photolyzed the iron complex **4** in THF in the presence of excess acrylonitrile at 67°, leading to C–C bond formation products **18** and **19** in a 6:1 ratio (*Scheme 4*). Presumably η^3-allyl complex **17** is the intermediate which reacts with acrylonitrile at the terminal end of the allylic system.

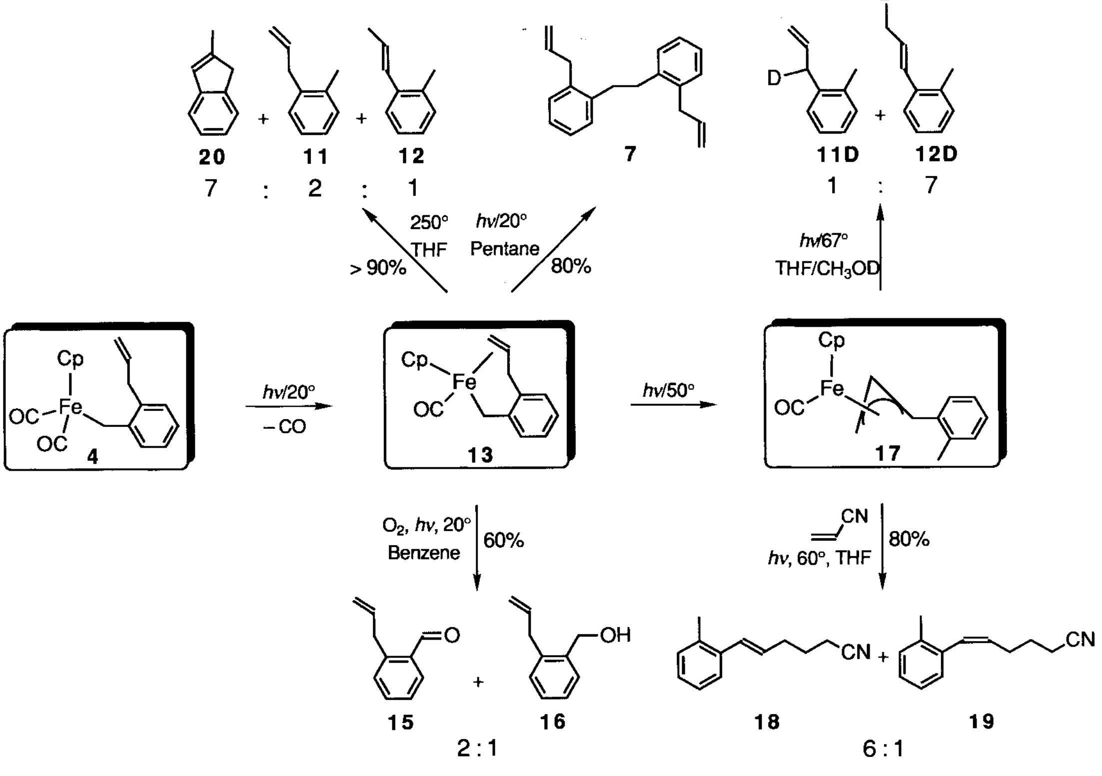
Scheme 4



Thermal decomposition of a THF solution of **4** in a GC injector at 200° gave no detectable amounts of organic compounds, whereas π-olefin complex **13** led at 250° to the formation of methylindene **20**, allylbenzene **11**, and styrene derivative **12** in a 7:2:1 ratio. Thermolysis of neat **13** gave, within experimental error, an identical product mixture. Thus, the thermal reaction of **13** results in both homolysis of the Fe–C bond and decomplexation of the olefin. Methylidene **20** could be formed *via* intramolecular radical cyclization [3] followed by β-elimination [6] and C=C bond isomerization.

3. Conclusion. – *Scheme 5* illustrates the reactions of the iron complexes **4**→**13**→**17**. Photolysis of *o*-allylbenzyliron complex **4** at 20° yields π complex **13**. Further irradiation at 50° leads to η^3-allyl complex **17**. Both reactions occur within a few min. The π complex **13** can be trapped by O_2 at 20° *via* photolysis, leading to aldehyde **15** and alcohol **16**.

Scheme 5



Photolysis of **13** at 20° gives the metal-free dimer **7**. By heating the π complex **13** to 250°, cyclization product **20** is formed. The π-allyl complex **17** can also be trapped. Thus, photolysis in the presence of acrylonitrile yields products of C–C bond formation **18** and **19**, and reaction with CH_3OD gives deuterated products **11D** and **12D**.

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Experimental Part

General. THF was freshly distilled from potassium benzophenone. Benzene was distilled from CaH_2 and stored over molecular sieves. All reactions and manipulations of organometallic reagents were carried out under Ar. The irradiation experiments were carried out with an *Osram Power Star HQI T 250/D* sunlamp. Chromatography utilized silica gel: *C 560KV* 35–70 µm, *Chemische Fabrik Uetikon*, or aluminium oxide: *507C* neutral, *Fluka*, activity III. GC: *Carlo Erba 6000* with flame-ionization detector coupled to a *Shimadzu C-R4A* integrator (conditions: 25 m *OV-1701* or 25 m *SE-30*, 50° to 260° at 10°/min). M.p.: uncorrected. IR: *Perkin Elmer 781* spectrophotometer. ^{1}H- and ^{13}C-NMR: either a *Varian Gemini 300* or a *Varian VXR 400* (TMS as internal standard). MS: either *VG 70-250* or *Varian MAT 212*. GC/MS: *Hewlett-Packard 5790A* gas chromatograph coupled to a *Hewlett-Packard 5970A* mass-selective detector (conditions; 25 m *SE-30*, 50° to 260° at 10°/min).

(2-Allylbenzyl)dicarbonyl(η^5-cyclopentadienyl)iron (**4**). *2*-Allylbenzyl chloride (**6**; 5.50 g, 33.0 mmol) was added dropwise at 20° to a soln. of sodium dicarbonyl(η^5-cyclopentadienyl)ferrate (**5**; 33.9 mmol) in THF (60 ml). After 2 h, the solvent was removed. The brown residue was diluted with pentane (200 ml), filtered, and purified by chromatography on aluminium oxide (pentane). The yellow band was collected. Removal of the solvent gave 5.00 g (49%) of **4** as yellow crystals. M.p. 41–42°. IR (KBr): 1985, 1920. ^{1}H-NMR (300 MHz; C_6D_6): 2.86 (*s*, FeCH_2Ar); 3.65 (br. *d*, J = 6.4, ArCH_2CH=CH$_E$H$_Z$); 4.11 (*s*, C_5H_5); 5.23 (br. *d*, J = 10.0, ArCH$_2$CH=CH_EH$_Z$); 5.26 (br. *d*, J = 17.0, ArCH$_2$CH=CH$_E$$H_Z$); 6.25 (*dddd*, J = 17.0, 10.0, 6.4, 6.4, ArCH$_2$C*H*=CH$_E$H$_Z$); 7.08–7.53 (*m*, Ar). ^{13}C-NMR (75.5 MHz; $CDCl_3$): 0.4 (Fe*C*H$_2$Ar); 37.4 (Ar*C*H$_2$CH=CH$_2$); 85.5 (C_5H_5); 115.5 (ArCH$_2$CH=*C*H$_2$); 123.7 (C(5)); 126.3 (C(4)); 128.9 (C(6)); 129.4 (C(3)); 135.3 (C(2)); 138.0 (ArCH$_2$*C*H=CH$_2$); 151.0 (C(1)); 217.2 (2 CO). MS (FB+): 309 ($[M + 1]^+$), 308 (M^+), 252. Anal. calc. for $C_{17}H_{16}FeO_2$ (308.16): C 66.24, H 5.23; found: C 66.06, H 5.31.

[η^2-(2-Allylbenzyl)]carbonyl(η^5-cyclopentadienyl)iron (**13**). A soln. of **4** (150 mg, 0.49 mmol) in CH_2Cl_2 (19 ml) was photolyzed at 20° for 7 min. The solvent was removed. The brown residue was diluted with pentane/Et_2O 2:1 (50 ml), filtered, and purified by chromatography on aluminium oxide (pentane). The orange band was collected. Removal of the solvent gave 130 mg (95%) of **13** as red crystals. M.p. 90°. IR (KBr): 1947. ^{1}H-NMR (300 MHz; C_6D_6): 1.31 (*s*, J = 13.1, ArCH$_\alpha$H$_\beta$–CH=CH$_E$$H_Z$); 1.66 (*d*, J = 8.2, Fe–CH_αH$_\beta$–Ar); 2.69 (*dd*, J = 14.8, 4.1, ArCH_αH$_\beta$–CH=CH$_E$H$_Z$); 2.78 (*d*, J = 8.2, FeCH$_\alpha$$H_\beta$–Ar); 3.00 (*d*, J = 8.1, ArCH$_\alpha$H$_\beta$–CH=CH_EH$_Z$); 3.22 (*dd*, J = 14.8, 4.2, ArCH$_\alpha$$H_\beta$–CH=CH$_EH_Z$); 3.95 (*s*, C_5H_5); 4.48 (*dddd*, J = 13.1, 8.1, 4.2, 4.1, ArCH$_\alpha$H$_\beta$–C*H*=CH$_E$H$_Z$); 6.80–7.25 (*m*, Ar). ^{13}C-NMR (75.5 MHz; $CDCl_3$): 1.0 (Fe*C*H$_2$Ar); 5.5 (ArCH$_2$CH=*C*H$_2$); 37.4 (ArCH$_2$CH=*C*H$_2$); 34.0 (Ar*C*H$_2$CH=CH$_2$); 37.9 (ArCH$_2$*C*H=CH$_2$); 84.8 (C_5H_5); 122.5 (C(5)); 124.2 (C(4)); 126.2 (C(6)); 127.8 (C(3)); 138.0 (C(2)); 150.1 (C(1)); 224.8 (CO). MS (FB+): 280 (M^+), 252. Anal. calc. for $C_{16}H_{16}FeO$ (280.06): C 68.60, H 5.76; found: C 68.32, H 5.48.

Carbonyl(η^5-cyclopentadienyl){η^3-[3-(2-methylphenyl)alkyl]}iron (**17**). A soln. of **13** (5 mg, 0.02 mmol) in C_6D_6 (0.7 ml) was irradiated with a sunlamp at 50°. After 30, 60, and 120 s, ^{1}H-NMR-spectra were recorded. The signals of **13** decreased, while the signals of **17** increased in intensity. ^{1}H-NMR (300 MHz; C_6D_6): *Table 2*.

Photolysis of **4** *at 20° in Pentane. 1,2-Bis(2-allylphenyl)ethane* (**7**). A soln. of **4** (200 mg, 0.65 mmol) in pentane (30 ml) was irradiated. After 8 h, the solvent was removed and the residue purified by chromatography on aluminium oxide (pentane). This gave 135 mg (80%) of **7** as a colorless oil. ^{1}H-NMR (400 MHz; $CDCl_3$): 2.88 (*s*, ArCH_2CH_2Ar); 3.39 (*d*, J = 6.3, 2 ArCH_2CH=CH$_E$H$_Z$); 4.99 (br. *dq*, J = 17.1, 2 ArCH$_2$CH=CH$_E$$H_Z$); 5.06 (br. *d*, J = 10.1, 2 ArCH$_2$CH=C$H_E$$H_Z$); 5.97 (*ddt*, J = 17.1, 10.1, 6.3, 2 ArCH$_2$C*H*=CH$_E$H$_Z$); 7.10–7.35 (*m*, 2 Ar). ^{13}C-NMR (101 MHz; $CDCl_3$): 34.2 (2 Ar*C*H$_2$CH=CH$_2$); 37.0 (Ar*C*H$_2$*C*H$_2$Ar); 115.8 (2 ArCH$_2$CH=*C*H$_2$); 126.3 (2 C(5)); 126.5 (2 C(4)); 129.3 (2 C(6)); 129.7 (2 C(3)); 137.3 (2 ArCH$_2$*C*H=CH$_2$); 137.6 (2 C(1)); 140.0 (2 C(2)). MS: calc. for $C_{20}H_{22}$: 262.1722; found: 262.1739.

Photolysis of **4** *at 20° in the Presence of O_2. 2-Allylbenzaldehyde* (**15**), *2-Allylbenzyl Alcohol* (**16**). Dry O_2 was bubbled through a soln. of **4** (200 mg, 0.65 mmol) in benzene (20 ml), while the soln. was irradiated for 6 h at 20°. The solvent was removed and the residue purified by chromatography on aluminium oxide (pentane/Et_2O 10:1) to yield 80 mg (60%) of **15** and **16** in a 2:1 ratio.

Data of **15** [7]: ^{1}H-NMR (300 MHz; $CDCl_3$): 3.83 (*d*, J = 6.0, ArCH_2CH=CH$_E$H$_Z$); 5.00 (*dd*, J = 17.1, 1.5, ArCH$_2$CH=CH$_E$$H_Z$); 5.10 (*dd*, J = 10.0, 1.5, ArCH$_2$CH=CH_EH$_Z$); 6.04 (br. *ddt*, J = 17.0, 10.0, 6.2, ArCH$_2$C*H*=CH$_E$H$_Z$); 7.40–7.86 (*m*, Ar); 10.27 (*s*, ArC*H*=O). GC/MS: 146 (M^+), 145, 131, 118.

Data of **16**: [3]. ^{1}H-NMR (300 MHz; $CDCl_3$): 1.59 (*s*, ArCH$_2$O*H*); 3.41 (*dt*, J = 6.2, 1.6, ArCH_2CH=CH$_E$H$_Z$); 4.78 (*s*, ArCH_2OH); 5.03 (*dt*, J = 17.0, 1.6, ArCH$_2$CH=CH$_E$H$_Z$); 5.10 (*dt*, J = 10.0, 1.6, ArCH$_2$CH=CH_EH$_Z$); 6.03 (br. *ddt*, J = 17.0, 10.0, 6.2, ArCH$_2$C*H*=CH$_E$H$_Z$); 7.22–7.43 (*m*, Ar). GC/MS: 148 (M^+), 131, 130, 115.

Photolysis of **4** *at 67° in THF. 2-Methylidene* (**20**) [8], *1-Methyl-2-(prop-2-enyl)benzene* (**11**) [9], *and 1-Methyl-2-(prop-1-enyl)benzene* (**12**) [9]. A soln. of **4** (300 mg, 0.97 mmol) in THF (20 ml) was irradiated at 67° for 12 h. The solvent was removed and the residue purified by chromatography on silica gel (pentane). A mixture of 105 mg (82%) **20**, **11**, and **12** was isolated in a 1:11:89 ratio. Compound **20** was synthesized independently [8].

Data of **20**: ^{1}H-NMR (400 MHz; $\mathrm{CDCl_3}$): 2.14 (*s*, $\mathrm{CH_3}$); 3.28 (br. *s*, $\mathrm{ArC}H_2$); 6.47 (br. *s*, CH=C); 7.10–7.40 (*m*, Ar). EI-MS: 130 (M^+), 129, 115.

Data of **11**: ^{1}H-NMR (400 MHz; $\mathrm{CDCl_3}$): 2.28 (*s*, $\mathrm{CH_3}$); 3.36 (*dt*, J = 6.4, 1.6, $\mathrm{ArC}H_2\mathrm{CH{=}CH_ZH_E}$); 4.99 (*dq*, J = 17.1, 1.7, $\mathrm{ArCH_2CH{=}C}H_Z\mathrm{H}_E$); 5.05 (*dq*, J = 10.1, 1.7, $\mathrm{ArCH_2CH{=}CH}_Z H_E$); 5.95 (*ddt*, J = 17.1, 10.1, 6.4, $\mathrm{ArCH_2C}H\mathrm{{=}CH_ZH_E}$); 7.10–7.40 (*m*, Ar). EI-MS: 132 (M^+), 117, 115, 91.

Data of **12**: ^{1}H-NMR (400 MHz; $\mathrm{CDCl_3}$): 1.90 (*dd*, J = 6.6, 1.6, $\mathrm{ArCH{=}CHC}H_3$); 2.32 (*s*, $\mathrm{ArC}H_3$); 6.10 (*dq*, J = 15.7, 6.6, $\mathrm{ArCH{=}C}H\mathrm{{-}CH_3}$); 6.59 (*dq*, J = 15.6, 1.6, $\mathrm{ArC}H\mathrm{{=}CHCH_3}$); 7.10–7.40 (*m*, Ar). EI-MS: 132 (M^+), 117, 115, 91.

Photolysis of **4** *at 67° in the Presence of* CH_3OD *1-Methyl-2-([1-$^2H_1$]prop-2-enyl)benzene* (**11D**) *and 1-Methyl-2-([3-$^2H_1$]prop-1-enyl)benzene* (**12D**). A soln. of **4** (20 mg, 0.065 mmol) in THF (5 ml) and $\mathrm{CH_3OD}$ (0.5 ml) was irradiated at 67° for 12 h. The solvent was removed and the residue purified by chromatography on silica gel (pentane). A mixture of 7 mg (90%) of **11D** and **12D** was isolated in a 1:7 ratio.

Data of **11D**: ^{1}H-NMR (400 MHz; $\mathrm{CDCl_3}$): 2.29 (*s*, $\mathrm{ArC}H_3$); 3.39 (*dtt*, J = 6.4, 2.0, 1.7, $\mathrm{ArC}H\mathrm{D{-}CH{=}CH_ZH_E}$); 4.99 (br. *d*, J = 17.2, $\mathrm{ArCH_2CH{=}C}H_Z\mathrm{H}_E$); 5.05 (br. *d*, J = 10.3, $\mathrm{ArCH_2CH{=}CH}_Z H_E$); 5.95 (*m*, $\mathrm{ArCH_2C}H\mathrm{{=}CH_ZH_E}$); 7.10–7.40 (*m*, Ar). EI-MS: 133 (M^+), 132, 118, 117, 116, 115, 92, 91.

Data of **12D**: ^{1}H-NMR (400 MHz; $\mathrm{CDCl_3}$): 1.89 (br. *d*, J = 6.6, $\mathrm{ArCH{=}CHC}H_2\mathrm{D}$); 2.33 (*s*, $\mathrm{ArC}H_3$); 6.10 (*dtt*, J = 15.6, 6.6, 1.5, $\mathrm{ArCH{=}C}H\mathrm{CH_2D}$); 6.59 (br. *dt*, J = 15.6, 1.5, $\mathrm{ArC}H\mathrm{{=}CHCH_2D}$); 7.10–7.40 (*m*, Ar). EI-MS: 133 (M^+), 132, 118, 117, 116, 115, 92, 91.

Photolysis of **4** *at 67° in the Presence of Acrylonitrile. (*E*)-6-(2-Methylphenyl)hex-5-enenitrile* (**18**) *and (*Z*)-6-(2-Methylphenyl)hex-5-enenitrile* (**19**). A soln. of **4** (200 mg, 0.65 mmol) and acrylonitrile (690 mg, 13.0 mmol) in THF (20 ml) was irradiated at 67° for 3 h. The solvent was removed and the residue purified by chromatography on aluminium oxide (pentane/$\mathrm{Et_2O}$ 10:1). A mixture of 97 mg (80%) **18** and **19** was isolated in a 1:6 ratio.

Data of **18**: IR (Film): 2250. ^{1}H-NMR (300 MHz; $\mathrm{CDCl_3}$): =1.87 (*quint.*, J = 7.2, $\mathrm{CH_2C}H_2\mathrm{CH_2CN}$); 2.28 (br. *q*, J = 7.2, $\mathrm{CH{=}CHC}H_2\mathrm{CH_2}$); 2.35 (*s*, $\mathrm{ArC}H_3$); 2.42 (*t*, J = 7.2, $\mathrm{CH_2C}H_2\mathrm{CN}$); 6.00 (*dt*, J = 15.6, 7.2, $\mathrm{ArCH{=}C}H\mathrm{CH_2}$); 6.68 (*d*, J = 15.6, $\mathrm{ArC}H\mathrm{{=}CHCH_2}$); 7.12–7.43 (*m*, Ar). ^{13}C-NMR (101 MHz; $\mathrm{CDCl_3}$): 16.5 ($\mathrm{CH_2CH_2}C\mathrm{H_2CN}$); 19.8 ($\mathrm{Ar}C\mathrm{H_3}$); 25.1 ($\mathrm{CH_2}C\mathrm{H_2CH_2CN}$); 32.0 ($C\mathrm{H_2CH_2CH_2CN}$); 119.5 (CN); 125.5, 126.6, 127.3, 129.0, 130.0, 130.3, 135.1, 136.3 (6 arom. C, $C\mathrm{H{=}}C\mathrm{H}$). EI-MS: 185 ($M^+$), 184, 131, 129, 115, 91.

Data of **19**: ^{1}H-NMR (300 MHz; $\mathrm{CDCl_3}$): 1.77 (*quint.*, J = 7.8, $\mathrm{CH_2C}H_2\mathrm{CH_2CN}$); 2.25 (*s*, $\mathrm{ArC}H_3$); 2.30 (*m*, $\mathrm{CH{=}CHC}H_2\mathrm{CH_2}$); 2.41 (*t*, J = 7.8, $\mathrm{CH_2C}H_2\mathrm{CN}$); 5.67 (*dt*, J = 11.5, 7.3, $\mathrm{ArCH{=}C}H\mathrm{CH_2}$); 6.56 (br. *d*, J = 11.5, $\mathrm{ArC}H\mathrm{{=}CHCH_2}$); 7.12–7.43 (*m*, Ar). MS: calc. for $\mathrm{C_{13}H_{15}N}$: 185.1205; found: 185.1195.

Thermolysis of (**13**). Under Ar, **13** (5 mg, 0.02 mmol) was heated at 250° for 3 h. After extraction with 5 ml $\mathrm{Et_2O}$ the products **20**, **11**, and **12** were detected by GC in a 7:2:1 ratio. In addition, traces of **7** were detected (< 1%). The products were characterized by comparison by GC/MS and ^{1}H-NMR with those of authentic samples.

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